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# PREPARATION OF COMPOSITE MATERIALS IN SPACE

VOLUME I - EXECUTIVE SUMMARY

FINAL REPORT

GENERAL DYNAMICS
Convair Aerospace Division

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# FOREWORD

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# SUMMARY


This document is the final technical report on the "Preparation of Composite Materials in Space" under Contract NAS8-27806. It gives an account of the performed investigations and their results. The report is presented in two volumes.

The objective of the study was to define promising materials, significant processing criteria and the related processing techniques and apparatus for the preparation of composites in space, and to establish a program for zero-g experiments and the required developmental efforts.

The study was directed at the preparation of the following composite types: 1) metal-base fiber and particle composites, including cemented compacts, 2) controlled density metals, comprising plain and reinforced metal foams, and 3) unidirectionally solidified eutectic alloys.

The materials and processing requirements for these composite types were defined by theoretical studies and laboratory investigations, comprising the evaluation of materials, processing techniques and individual processing parameters. These efforts included the establishment of the basic criteria for liquid-state processing by fluid mechanics studies and experiments, the compilation of applicable materials data, the definition of sample preparation techniques for zero-g experiments and the construction of special laboratory facilities.

A program of suborbital and orbital experiments for the 1972 to 1978 time period was established, identifying materials, processes and the required experiment equipment.

Some of the more significant conclusions reached in the study are:

1. The zero-g environment of orbital operations offers the capability to produce metal-base composite materials and castings which exhibit properties and, particularly, unique combinations of properties that cannot be achieved in terrestrial production.

2. The primary criterion for effective fiber/particle composites and controlled density materials is the achievement of perfect random dispersion. Due to the complexity of interactions between molten metals and solid or gaseous dispersions, material treatments and processing techniques have to be developed specifically for each material combination.

3. The behavior of mixtures of liquid metals and properly treated fibers or particles can be accurately predicted for zero-g conditions or specific low-g levels. Relationships and data for such predictions have been established theoretically and experimentally.

4. Controlled density materials can be produced either by the dispersion of gas in the molten metal or by decomposition of pre-dispersed solid particles. The latter method, for which specific materials have been defined experimentally, is most adaptable to initial zero-g experiments.

5. The primary requirements for the preparation of unidirectional eutectics are highest material purity and extremely low solidification rates.

6. Since the concerned composites are unique to zero-g processing, conclusive property data can only be obtained from zero-g experiments. With the exception of unidirectional eutectics, which call for sustained zero-g conditions, adequate data can be generated in suborbital experiments. For highest experiment return at minimum cost, efforts should be concentrated on one base material for each composite type, and the evaluation of all significant variables.

## SECTION 1

## OBJECTIVES AND SCOPE

The general objective of this study was to evaluate the potential of producing new composite materials by liquid-state processing in space, unfeasible in the terrestrial environment due to segregation. The specific objectives were:

a. To define the most promising metal-base composite materials for processing in zero-g.

b. To define the materials and processing parameters for the preparation of typical composites in zero-g.

c. To identify major problem areas and to investigate them in sufficient depth to outline an approach for the solution of these problems.

d. To define experimental programs required for the solution of problems identified in (c) in ground-based studies and experiments, and in zero-g experiments.

e. To define process techniques, peculiar apparatus concepts, and operational requirements for experimental investigations in orbital facilities.

The composite classes investigated in this study comprise: 1) Fiber- and Particle-Reinforced Metal-Base Composites, 2) Controlled Density Metals (plain and reinforced metal foams), and 3) unidirectional eutectics. Main emphasis has been placed on composite classes 1) and 2) in view of the wide variety of individual composite types, the high applications potential and the uniqueness of the processes to zero-g.

Originally, the scope identified two other classes: Cemented Compacts and Supersaturated Alloys. However, Cemented Compacts can be considered as a specific type of particle composites and have, therefore, been included in class 1); the investigation of supersaturated alloys was deleted in view of a separate study of this subject under another contract.

To accomplish the stated objectives, the following scope of major subject areas was adopted for the study.

a. Conceptual evaluation of composites and preliminary definition of processes, materials, and their predominant criteria for the purpose of identifying the subjects for detailed study.

b. Establishment of relationships and numerical data for the behavior of liquids and mixtures by theoretical studies and laboratory experiments.

c. Experimental investigation of processing parameters that govern the dispersion of solids and gases in liquid metals.

d. Experimental investigation of surface requirements and treatments to achieve high wettability of reinforcements.

e. Analysis of the compatibility of reinforcements with liquid metals and definition of compatible material combinations.

f. Assessment of attainable composite properties, as related to materials, reinforcement configuration, and reinforcement content.

g. Experimental determination of reinforcement content limitations as related to reinforcement configuration and mold size.

h. Evaluation of methods for the generation of metal foams and experimental investigation of the most promising methods.

i. Theoretical analysis and laboratory experiments on the unidirectional solidification of eutectic alloys.

j. Compilation of applicable materials data and measurement of properties not reported in the literature.

k. Experimental preparation and evaluation of composite samples and definition of techniques for the preparation of zero-g experiment samples.

l. Definition of the most effective materials and processing methods for initial zero-g experiments.

m. Study of process and apparatus requirements for experiments in suborbital and orbital facilities.

Laboratory investigations further necessitated the development of special experimental facilities. Major facilities constructed in the course of the study include several apparatus for the measurement of liquid-metal properties, an apparatus for the measurement of segregation rate and dispersion phenomena, an apparatus for the melting and solidification profiles of specific sample configurations, an induction furnace for processing of eutectic alloys, and a high-purity argon chamber for material preparation and sample assembly, equipped with all necessary tooling.

## SECTION 2

## SUMMARY OF RESULTS

### 2.1 FIBER AND PARTICLE COMPOSITES

The necessity of a low- or zero-g environment for the preparation of composites by processing in the liquid-matrix state, or the uniqueness as a space process, was predicated on the assumption of a prohibitive segregation in the 1-g environment. The degree of segregation encountered in 1-g has never been defined numerically. Theoretical studies and extensive laboratory experiments were carried out on the segregation rates and particle mobility as related to particle configuration, matrix viscosity, density difference and g-level. It was found that even at a density difference as low as 0.1 g/cm$^3$, the 1-g segregation rate for the viscosity range of molten metals is still in the order of 0.25 cm/sec. It was further found that the segregation rate is approximately proportional to the g-level; i.e., that a reduction of the g-level by several orders of magnitude is matched by a reduction of the segregation rate by equal orders of magnitude. It was concluded that liquid-state composite production and composite casting are unfeasible in the terrestrial environment and, consequently, unique to space processing.

2.1.1 <u>DISPERSION CRITERIA.</u> The prime requirement for the achievement of a castable random composite with anisotropic properties is the uniform dispersion of reinforcements. It calls for two basic reinforcement characteristics: 1) wettability in the liquid matrix, and 2) freedom from adhering gases. Wetting characteristics have been found to be extremely sensitive to changes in surface conditions, such as the presence of minute oxide films. The sensitivity varies with specific material combinations and cannot be generalized.

Unfortunately, the most attractive reinforcement materials, such as graphite, boron, tungsten or whiskers are nonwetting with regard to practically all useful matrix metals and coating materials and techniques have to be devised to achieve wettability. Coating experiments have further shown that conventional thin-film coatings are readily dissolved in most liquid metals, and that the coating thickness has to be adapted to specific matrix metals to assure stability for the duration and temperature of the liquid-state processing cycle.

2.1.2 <u>COMPOSITE PREPARATION.</u> The preparation of the composite consists of two operations: 1) joining of the reinforcements with the molten matrix, and 2) mixing to achieve dispersion. The extent to which these operations are carried in zero-g or in 1-g depends on the method of composite preparation. Three methods

have been defined:

a. Terrestrial "dry" mixing and compaction of well prepared matrix granules or powder and reinforcements; the dispersion is established in 1-g while the zero-g process involves only the joining operation in the form of a matrix-melting cycle.

b. Terrestrial joining of the component materials into an ingot, containing the reinforcements in segregated position. The zero-g process comprises re-melting of the ingot and the establishment of dispersion by dynamic mixing.

c. Performance of all processing phases in zero-g.

A successful joining of molten matrix and reinforcements (immersion) calls for 1) careful cleaning of reinforcements and other surface treatments, such as precoating with matrix material, and 2) an immersion technique which prevents the infiltration of gases into the mixture. Individual experiments and sample preparations carried out under various environmental conditions indicate the necessity to perform the entire sequence of processing steps either in a high-purity argon enclosure or in high vacuum, depending on the base material.

All reinforcement treatments, joining and dispersion techniques vary extensively with the concerned materials and have to be developed individually and in considerable detail for each specific material combination.

2.1.3 MATRIX-REINFORCEMENT BOND STRENGTH. An effective composite further calls for a high bond strength between matrix and reinforcements after soldification. It has been found that adhesion and bond strength are not necessarily related to wetting characteristics, and material data can only be obtained experimentally. Pertinent testing techniques have been developed and demonstrated. Studies have further indicated that the bond strength can, in specific material combinations, be increased substantially by diffusion heat treatment of the solidified composite.

2.1.4 MATERIALS SELECTION. The selection of effective reinforcement materials was based on three criteria: 1) high strength, 2) no strength degradation at the matrix melting temperature, and 3) chemical compatibility with the liquid matrix. For moderate temperature matrices (to 800°C) a wide variety of reinforcements have been identified which fulfill these requirements, such as graphite fibers, chopped tungsten wire, boron filaments, and several whiskers (SiC, $Al_2O_3$, BN). Composites of these materials with an aluminum or magnesium alloy matrix are very promising.

Primary matrix candidates for higher temperatures are Ni, Co, and Fe; however, at the concerned liquid-matrix temperatures only $Al_2O_3$ fulfills all reinforcement requirements. For graphite fibers and whiskers of SiC or $Si_3N_4$ appropriate coatings have to be found to prevent chemical reaction. Boron and BN are ineffective due to strength degradation.

2.1.5 REINFORCEMENT CONFIGURATION AND CONTENT. The configuration of the individual reinforcements depends on available sizes and the product requirements. The prime applications of particles are refined-grain castings or dispersion-stabilized materials, both for increased creep resistance; the required particle contents are in the order of one percent. For high-strength particle composites, higher contents are required with a geometrical limitation of 60%.

High-strength fiber or whisker composites call for high reinforcement L/D (over 50) and highest reinforcement content. While high L/D fibers and whiskers are readily available, the maximum content of a perfectly random-oriented high L/D mixture is limited to 14%.

2.1.6 CONCLUSIONS. On the basis of these results, three major conclusions have been drawn:

1. The production of composites by liquid-matrix processing is unique to the zero-g environment due to the prohibitive segregation effects in 1-g processing. By the same token, conclusive data on the composite capabilities and their relation to processing parameters can only be obtained in zero-g experiments.

2. The necessary detailed processing parameters and techniques, particularly those related to reinforcement preparation and dispersion, have to be developed specifically for each base metal.

3. The most promising base materials for initial zero-g experiments are those with moderate melting temperature, such as Al and Mg.

## 2.2 CONTROLLED DENSITY METALS (METAL FOAMS)

Controlled density materials are essentially refined metal foams obtained by the dispersion of gas in the form of discrete microbubbles in the molten matrix and solidification. In view of the low viscosity of molten metals (in the order of water) and the high density differences, liquid/gas mixtures are extremely sensitive to gravity and exhibit immediate segregation in the 1-g environment. The required mixture stability during liquid-state processing can only be obtained in zero-g or at very low g-levels. The production of controlled density metals is, therefore, a unique space manufacturing process.

2.2.1 MATERIAL TYPES AND PRODUCT CHARACTERISTICS. Controlled density metals have been classified into three major types: 1) plain metal foams, 2) long-fiber reinforced metal foams,3) short-fiber reinforced metal foams.

The prime characteristics of plain metal foams are high stiffness-to-density ratio, high damping capability, high impact resistance, and low thermal conductivity.

The second type of metal foam is reinforced with high-strength fibers whose length is a multiple of the average gas bubble diameter. While the reduction of bulk density is considerably less than in type 1, such reinforced foams are expected to exhibit a unique combination of high stiffness and strength-to-density ratio with high shock and impact resistance.

In the third material type, the length of the reinforcements is less than the average bubble diameter. Short-fiber reinforced foams combine the high bulk density reduction of type 1 with the mechanical properties of type 2, yet exhibit a higher degree of plastic deformation capability.

Potential applications of reinforced foams are aircraft components with high stiffness-to-density ratio, high damping coefficients and high impact resistance, thermostructural components, lightweight armor,and deep-sea components with high resistance to external pressures.

2.2.2 DISPERSION CRITERIA. Fundamental fluid-mechanics relationships were established for the behavior of gas bubbles in liquids under zero-g, the effect of thermal gradients, the interactions between individual bubbles,and the interaction with solid particles (fibers) and the container wall. The following requirements for dispersion were established and verified in laboratory experiments:

a. In the liquid-state processing of plain foams there is a strong tendency for bubble coalescence. This can be suppressed by the use of an appropriate gas, which reacts with the bubble surface and produces a stabilized gas/liquid interface.

b. In long-fiber reinforced foams, the gas dispersion is dictated by the geometry of the fiber framework, as the bubbles are trapped and embedded in the inter-fiber spaces. This trapping effect renders the composite less sensitive to g-forces and stable dispersion may be maintained at g-levels as high as $10^{-3}$g.

c. In short-fiber reinforced foams, the dispersion of fibers is governed by the bubble distribution. The fibers have the tendency to agglomerate at the bubble walls in parallel patterns, effectively enhancing the deformation resistance of the individual bubble and the bulk material.

d. In all reinforced foams, the fibers have to exhibit high wetting characteristics to be retained in the matrix and to assure complete matrix coverage of fibers located at bubble interfaces. Both effects are essential to maintain continuity of the composite matrix.

2.2.3 FOAM GENERATION METHODS. The following methods of foam generation have been identified: 1) dispersion of gas-filled microballoons; 2) gas generation by the thermal decomposition of additives, predispersed in the matrix in the form of a compact ("compact foaming"); 3) gas injection foaming; 4) ultrasonic foaming; and 5) nucleate foaming. Methods 3, 4, and 5 involve extensive equipment development. In contrast, methods 1 and 2 exhibit relative simplicity, yet at the same time permit the evaluation of all essential processing parameters and the integration in early zero-g experiments. Laboratory investigations were, therefore, limited to methods 1 and 2 and exploratory experiments on method 3.

2.2.4 MICROBALLOON FOAM EXPERIMENTS. Foam preparation with gas-filled microballoons may be accomplished either by liquid-state mixing, or by compacting and melting. A prerequisite of the first technique is temperature compatibility of the microsphere wall material with the molten matrix. Foam preparation by the compacting technique is limited by the pressure resistance of available microsphere materials, while the temperature stability is less significant.

A mandatory requirement is high wettability of the microspheres. While coating was accomplished successfully, it was found that the coating thickness has to be substantial in order to maintain wettability and dispersion integrity to the time of solidification.

2.2.5 COMPACT FOAMING EXPERIMENTS. The most attractive feature of the compact foaming technique is the generation of the gas and foam during the low-g processing cycle. It further does not require agitation since the dispersion is established in the solid-state compact preparation in 1-g. The process consists of the following operations:

a. Preparation (cleaning, deoxidation) of the granules of base material and foaming agents as well as fibers for reinforced foam.

b. Dosaging of component materials to obtain a specific foam density.

c. Solid-state ("dry") mixing of granules and, if applicable, reinforcements.

d. Compacting into expandable mold.

e. Zero-g processing: heating, melting and solidification.

Foaming agents were defined for low-melting metals, such as tin, and for the melting regime of Al and Mg. The amounts of gas evolution as related to temperature and time and the most effective time-temperature programs were defined experimentally for various types of urea, hydrides and oxalates.

Dry mixing and compacting experiments showed that the entire solid-state processing from the preparation of component material has to be carried out in a high-purity argon environment. An argon chamber for sample preparation was set up, equipped with all tooling necessary for the individual solid-state processing steps.

The attainment of evenly dispersed foams was verified in laboratory experiments with samples of various compositions. There is a high degree of assurance that under low-g conditions perfect foams with a uniform gas distribution will be obtained.

2.2.6 GAS INJECTION FOAMING EXPERIMENTS. In view of the extensive equipment requirements of gas injection in liquid metals, experiments were confined to the investigation of gas injection techniques with transparent simulation liquids. It was found that gas injection foaming is most effective in the form of a continuous process, in which gases are injected at a specific point (nozzle) into a flow of molten metal (or a metal/fiber mixture).

2.2.7 CONCLUSIONS. In view of the high segregation effect in liquid/gas mixtures under 1-g and, consequently, the inability to produce measurable foam quantities in laboratory experiments, low-g experiments should be carried out at the earliest possible time. For such experiments an effective and reliable foaming technique has been defined and verified which permits the evaluation of all essential material and processing parameters typical of metal foams.

## 2.3 UNIDIRECTIONAL EUTECTICS

In some eutectic alloys, a unidirectional rod-like pattern of the intermetallic phase can be obtained by progressive unidirectional solidification. The resulting composites, consisting of intermetallic filaments embedded on the base metal, exhibit a high unidirectional strength which remains stable close to the melting temperature. Such composites have been successfully produced in the laboratory. However, the mechanical properties obtained in 1-g are only 50% of the theoretically provided properties, due to g-induced disturbances during the solidification process. It is expected that properties approaching the theoretical values are obtained in zero-g processing. The products are highly attractive for applications where high unidirectional strength at high temperatures is required, such as gas turbine blades.

The primary criterion for an optimum composite microstructure is the solidification (cooling progression) rate, which is expected to be much lower in zero-g than in

1-g. Laboratory experiments have further shown that the process is extremely sensitive to alloy impurities and that highest purity is essential for successful composite preparation. Promising materials for high temperature applications are tantalum and niobium-base eutectic alloys; they require, however high processing temperatures (2400 - 2800° C). For the establishment of processing data, which can only be accomplished in zero-g experiments, eutectics with moderate melting temperature, such as Al-Cu and Al-Ni alloys may be used.

In view of the extensive time requirements, exact processing data and material capabilities can only be obtained in orbital experiments. However, suborbital experiments may be useful for the investigation of processing parameters and for the definition of orbital experiments.

## 2.4 MATERIAL PROPERTIES

An extensive literature search for applicable materials data was carried out and the data compiled in tabular form. They comprise: a) solid-state properties of matrix metals, b) solid state properties of reinforcements, c) liquid-state properties of metals and, d) properties of transparent liquids for simulation experiments.

It was found that available data on liquid metals, such as surface tension, viscosity and density, are very incomplete and unreliable. They are, however, essential for the development of liquid-state processing techniques. Some of the data were, therefore, measured in specially designed and constructed laboratory facilities. Experimental data were further generated on the wetting characteristics of various reinforcement materials in contact with molten metals and on the bond strength of several reinforcement-matrix metal combinations after solidification.

## 2.5 ZERO-G FLUID MECHANICS

Extensive theoretical studies and experimental investigations were carried out on the fluid mechanics of liquid metals and mixtures in zero- and low-g. For the experimental investigations, transparent simulation liquids of the liquid-metal viscosity regime were used exclusively in view of the observation difficulties with liquid metals.

An excellent agreement of theoretical relationships and experimental data was obtained for segregation rates and fiber mobility which permits the prediction of the behavior of mixtures at various g-levels. Theoretical studies further produced relationships for the behavior of mixtures and specific solutions for various motion modes which provide a basis for the effective development of mixing techniques and for the achievement of stable dispersion.

## 2.6 ZERO-G EXPERIMENT REQUIREMENTS

The necessity of zero- or low-g experiments for the determination of composite capabilities and optimized process parameters has been clearly established in the course of the study. An evaluation of the capabilities of various zero-g facilities showed that the use of short-time facilities, such as drop towers of the KC-135 research aircraft is limited to the evaluation of fundamental phenomena and processing details. For fiber/particle composites and for controlled density materials, conclusive materials and process data can be obtained in suborbital experiments (research rockets). The preparation of prototype products of these composite classes and high-temperature unidirectional eutectics can only be obtained in orbital facilities.

Emphasis was, therefore, placed on the definition of experiment requirements in suborbital and orbital facilities. Flow diagrams were established for various processing methods. Material quantities, operational requirements and typical apparatus designs were defined for each, suborbital and orbital experiments.

## SECTION 3

## CONCLUSIONS

### 3.1 GENERAL CONCLUSIONS

a. The preparation of composites by liquid-state processing is unfeasible in the terrestrial environment due to prohibitive segregation. It is, therefore, unique to orbital gravity environments. This implies further, that capability data can only be ontained by the processing of samples in zero- or low-g and that an early activation of facilities for extended low-g testing is mandatory.

b. Liquid-state processing of composites is a new field of technology that is faced with a complete absence of fundamental and technological information. The generation of such information, including data for liquid metals, is a prerequisite for an effective and minimum-effort development of such composites and the related zero-g processes.

c. Due to the complexity of interactions between molten metals and solid or gaseous dispersions, material treatments and processing techniques have to be developed specifically for each material combination. The magnitude of the involved efforts calls for an initial concentration on a limited number of base materials and processing methods.

d. Zero-g produced composites promise properties and, particularly, unique combinations of properties which cannot be achieved in terrestrial production.

### 3.2 FIBER AND PARTICLE COMPOSITES

a. The preparation of fiber- and particle composites by liquid state processing is unique to zero- or low-g. Experimental measurements of segregation rates as related to density difference, matrix viscosity and reinforcement configuration showed that even at a density difference as low as 0.1 $g/cm^3$ prohibitive segregation is encountered in 1-g. For this reason it is impossible to prepare measurable laboratory samples. The properties of composites can only be derived from low-g experiments, and earliest activation of facilities for extended low-g testing are indicated.

b. The production of composites in zero gravity may be accomplished by either of three basic techniques: 1) Pre-dispersion of component materials by dry-mixing and compacting in 1-g, followed by a matrix-melting cycle in zero-g. 2) Casting of segregated mixture of component materials in 1-g, followed by melting, mixing and solidification in zero-g. 3) Performance of all processing phases in zero-g with the exception of reinforcement coatings and treatments which can be carried out in 1-g.

c. A mandatory requirement for reinforcement dispersion is high wetting characteristics. All effective reinforcement materials have been found to be non-wetting with regard to all defined matrix materials. Chemically stable coatings and the related coating techniques have to be developed specifically for each matrix-reinforcement combination.

d. In view of the high sensitivity wetting characteristics to surface contamination and oxidation, the preparation of component materials and the assembly of test samples have to be carried out in high-purity inert gas or in high vacuum.

e. A second reinforcement requirement is physical and chemical stability at the liquid matrix temperature and in contact with the liquid matrix. All reinforcements are physically and chemically compatible with molten Al and Mg. For base metals of higher melting temperature, only $Al_2O_3$ reinforcements are fully compatible with Ni, Co and Fe; all other reinforcements are either physically or chemically unstable. Reinforcements which exhibit strength degradation, such as B or BN are unfit for high temperature composites. For all others, barrier coatings have to be developed which prevent chemical reaction with the molten matrix.

f. The dynamic characteristics of liquid/solid mixtures in zero-g, such as the mobility of reinforcements in molten metals, the response of reinforcements to induced liquid motion modes and the sensitivity to specific low-g levels can be accurately predicted. Applicable relationships have been established by theoretical fluid mechanics studies and laboratory experiments.

g. For the concerned random-reinforced composites, the maximum content for particles is 60% and for high L/D fibers 14%. The fiber content limitation permits only moderate strength improvements. However, substantial improvements can be achieved by the combined dispersion of fibers and particles. Further the prime attractiveness of zero-g produced fiber and particle composites is the anisotropy of properties, the castability of end-product shapes and the potential of unique property combinations.

h. Material preparation techniques and processing parameters have to be developed and specified individually for each matrix/reinforcement materials combination. In view of the substantial efforts involved in each case, it is indicated to concentrate near-term efforts on one base material, such as aluminum, including the determination of composite properties in low-g experiments.

## 3.3 CONTROLLED DENSITY MATERIALS

a. In view of the low viscosity of liquid metals, a stable dispersion of discrete gas bubbles in a molten metal, necessary for the preparation of metal foams, can only be achieved at very low g-levels. This places a severe limitation on laboratory investigations. Capabilities of metal foams and the related liquid-state processing parameters can only be verified in zero-g experiments.

b. On the basis of capability assessments and applications studies, three types of promising controlled density metals have been identified: 1) plain (non-reinforced) metal foams; 2) short-fiber reinforced foams (ratio of fiber length to bubble diameter <1; 3) long-fiber reinforced foams (ratio >1).

c. Promising methods of foam generation are: 1) disperion of gas-filled microspheres; 2) gas generation by pre-dispersed additives ("compact foaming"); 3) gas injection foaming; 4) ultrasonic foaming and 5) nucleate foaming. For near-term development and zero-g experiments, the compact foaming method is most effective with regard to relative simplicity, demonstrated successfulness and adaptability to the limitations of suborbital experiments.

d. The behavior of individual gas bubbles and their interaction with reinforcements during liquid-state processing, such as the sensitivity to g-forces and to thermal gradients, can be predicted; initial fluid-mechanics relationships have been established.

e. The prime problem of foam generation is bubble coalescence. This may be prevented or minimized by any or combinations of the following means: 1) stationary mixture (as in the case of compact foaming); 2) wrapping of the gas bubbles in a fiber network (long-fiber reinforced foam); 3) use of a slightly oxidizing gas (gas injection foaming); and 4) matrix alloys which exhibit a high-viscosity melting range.

f. For reinforced foams, fibers have to exhibit high wettability in order to be retained in the matrix.

g. Successful foaming agents for base-metals of intermediate melting temperature, such as Al or Mg, are $TiH_2$ or $ZrH_2$.

h. For plain and short-fiber reinforced foams, the achievement of gas contents in the order of 50% is reasonably assured; higher gas contents are feasible. The selection of gas and reinforcement content depends on specific product applications.

i. Promising applications are: 1) for plain foams, underwater high-buoyancy components. 2) For reinforced foams, components of high stiffness and to density ratio, lightweight armor or structures of low thermal conductivity. Selected combinations of these properties are particularly attractive for air, surface and deep-sea weapon systems.

j. The investigations indicate clearly that specific foaming methods, specific material treatments and specific processing parameters have to be defined individually for each base metal and reinforcement combination. The most effective approach, technically and economically, is, therefore, a concentration of efforts on one base material and one foaming method with the objective to obtain product capability data from processing experiments in suborbital low-g test facilities. The obtained results will provide a reliable basis for the selection of other base materials, the development of other foaming methods and the definition of product applications.

## 3.4 UNIDIRECTIONAL EUTECTICS

a. The feasibility of achieving high unidirectional properties in certain eutectic alloys by discrete and unidirectional formation of the intermetallic phase has been demonstrated. Properties obtained in 1-g are, however, far from the theoretically expected values due to gravity-induced disturbances. It is expected that substantially higher properties are obtained by processing in zero-g.

b. The essential processing requirements are: 1) exact definition of the solidification (progressive cooling) rate which will be lower in zero-g and 2) highest purity of the alloys. The definition of the solidification rate can only be obtained in zero-g experiments.

c. Since the process is of a metallurgical nature and does not involve dynamic effects, zero-g experiments and equipment requirements are comparatively simple and similar to those required for single-crystal experiments.

d. Due to the high processing time and temperature requirements, suborbital experiments are limited to small samples of Al-base eutectics and metallurgical evaluation. Exact processing data and product capabilities can only be obtained in orbital experiments. They should be carried out with practical high-temperature materials, such as Nb-C and Ta-C alloys, producing Nb-NbC and Ta-$Ta_2C$ alloys, producing Nb-NbC and Ta-$Ta_2C$ composites of substantial size.

# SECTION 4

# RECOMMENDATIONS

## 4.1 GENERAL RECOMMENDATIONS

For near-term developmental efforts with the objective of generating experimental data on composite capabilities, the following approach is recommended:

a. Initial concentration of efforts on one effective base material and one processing method for each composite class. Specifically, to use aluminum alloys as base materials for all composite classes, and the compact method for fiber/particle composites and controlled density metals.

b. All efforts should be directed at the preparation of zero-g experiment samples of sufficient size to permit evaluation of mechanical properties.

c. Earliest activation of a flight test program with suborbital rockets and preparation of flight apparatus for the processing of composite samples.

d. On the basis of the results of initial zero-g experiments, extension of the composite program to other base metals and other processing methods.

## 4.2 SPECIFIC RECOMMENDATIONS

For an effective implementation of the recommended general approach, the following tasks should be accomplished, initially for the selected single base material and processing method, and later for other materials and methods:

a. In-depth investigation of wetting characteristics and chemical effects between reinforcements and the molten matrix.

b. Bond strength between reinforcements and matrix after solidification and potential improvements by diffusion treatment.

c. Definition of coatings or treatments and related techniques which fulfill all requirements of a and b.

d. Development of mixing modes and techniques to achieve random dispersion of reinforcements and/or gases.

e. Definition of optimum composite compositions with regard to expected composite properties and matrix microstructure.

f. Definition of gases for specific matrices which suppress bubble coalescence by surface stabilization.

g. Preparation of optimized gas-forming additives for compact foaming.

h. Establishment of detailed processing specifications for low-g experiments.

i. Establishment of techniques and facilities for the preparation of flight samples

j. Definition of methods for flight sample evaluation.

k. Correlation of predicted and experimental composite properties and definition of means for improvement.

l. Assessment of product applications on the basis of zero-g experiment results.

## 4.3 RECOMMENDED SUPPORTING EFFORTS

For an effective accomplishment of the technological efforts defined in 4.2, the performance of the following supporting studies is indicated:

a. Fundamental studies and experiments on the fluid mechanics of liquid metals and mixtures with solids and/or gases in zero-g, with emphasis on effective mixing modes and the achievement of perfect dispersion.

b. Effect of liquid metal properties, such as surface tension, viscosity and wetting characteristics upon dispersion and mixture stability.

c. Theoretical correlation of intermolecular forces and surface energies with the properties of liquid metals; application to the characteristics of liquid/solid and liquid/gas interfaces.

d. Experimental establishment of data for liquid metals, not available in the literature.

e. Assessment of the effect of small g-forces and thermal gradients upon composites during liquid-state processing and correlation with the results of zero-g experiments.

f. Establishment of analytical methods for the prediction of the bulk properties of random-reinforced composites and controlled density metals.

g. Analysis of the deformation mechanism and the limiting criteria of plain foams and fiber-reinforced composites under various loading modes; assessment of the performance of composites in specific product applications.

h. Definition of other than mechanical properties attainable in the concerned composites; definition of unique property combinations and promising product applications.

i. Investigation of individual processing parameters and zero-g phenomena, such as interface or solidification effects, in drop tower experiments.

## 4.4 RECOMMENDED EXPERIMENTAL PROGRAM

It has been clearly established in this study that a conclusive verification of composite capabilities and the pertinent processes can be obtained only in experiments under

zero- or low-g conditions. Orbital experiments involve high cost and long lead times. Consequently, an experimental program is recommended that provides for an early generation of urgently needed data and a gradual build-up of capabilities at minimum cost. It consists of the following sequence of experiment phases:

a. Laboratory experiments for the exact specification of zero-g experiments.

b. Experiments in drop towers and the KC-135 in support of a.

c. Extended low-g time experiments in research rockets to produce samples that can be tested for composite properties.

d. Experiments in automated satellites to accommodate larger material quantities and higher processing temperatures, as well as for the evaluation of product casting techniques.

e. Experiments in shuttle-based orbital laboratories for the production of prototype products.

An overview of the recommended experiment program is presented in the form of a master plan, Figure 4-1.

4.4.1 LABORATORY EXPERIMENTS. The prime purpose of laboratory experimentation is the definition of zero-g experiments in terms of materials and process specifications. It is recommended to devote laboratory work during the first half of CY 73 exclusively to the exact specification of Al-base rocket experiments, specifically the definition of composite compositions, sample configurations, processing specifications, and in-flight measurements, as well as techniques and procedures for sample preparation and evaluation. During the second half of 73, laboratory experiments on other matrix materials and on advanced processing methods should be started, which would continue through the following years as required. Laboratory work during the latter period would also include experiments in support of the evaluation of flight samples.

4.4.2 DROP TOWER AND KC-135 EXPERIMENTS. Short-time low-g experiments have two objectives: 1) evaluation of individual phenomena and processing details; 2) preparation of small feasibility demonstration samples. During the first half of CY 73 it is recommended to continue the bubble interface and the gas injection foaming experiments started in CY 72, and to prepare small (flat) foam samples by the compact foaming method with several low-melting alloys. During the second half of CY 73, experiments may be carried out on the metallurgical effect of various particle and fiber mixtures and on the effect of various mixing modes upon dispersion of reinforcements. Drop tower and/or KC-135 experiments in support of composite development and fundamental studies may be continued through the following years as indicated.

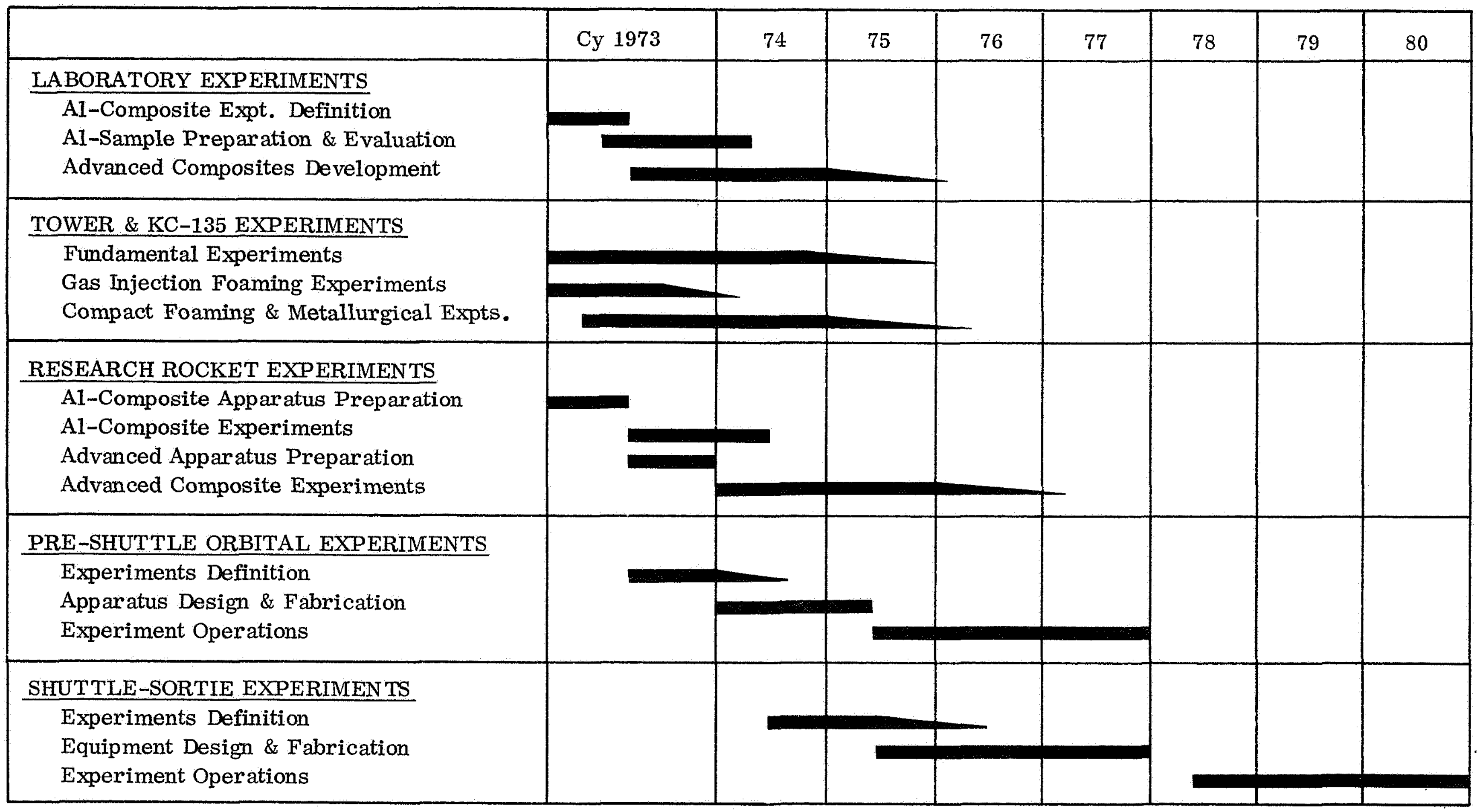


Figure 4-1. Experiment Program Master Plan 1973 — 1980

4.4.3 RESEARCH ROCKET EXPERIMENTS. It is strongly recommended to activate a research rocket program at the earliest possible time since it is a comparatively inexpensive means to obtain a conclusive feasibility and capability verification for composites, as well as for other space manufacturing processes. Assuming that flight opportunities become available in CY 73, work should be started immediately on apparatus design and fabrication, so that experiments can be carried out in the second part of CY 73. A minimum of three flights are recommended for CY 73 with Al-base fiber/particle composites and controlled density materials, which can be carried out in the same apparatus and would yield 10 samples. One flight is recommended for Al-base unidirectional eutectics, which may be combined with single-crystal experiments.

During the second half of CY 73, apparatus modifications should be defined and prepared to facilitate experiments with base metals of higher melting temperature and other processing methods. A high-frequency rocket test program is recommended for CY 74 (FY 74) to optimize composite compositions and to generate the data necessary for the definition of specific product applications. Rocket experiments may be continued throughout the subsequent years for the evaluation of new space manufacturing concepts that will undoubtedly evolve from present efforts.

4.4.4 AUTOMATED SATELLITES. It is recommended to program experiments on automated (unmanned) satellites for the 1975 - 1977 period. The long zero-g times of such vehicles that can be deployed with existing launch systems permit large material quantities, high processing temperatures, and verification of casting techniques representative of specific product requirements. Preliminary experiment definitions for the purpose of specifying equipment requirements should be made early in CY 73, followed by equipment design studies in the second half of CY 73. The definition of a detailed experiment program and the preparation of equipment hardware should be carried out in CY 74 to permit the start of the flight test program in CY 75.

4.4.5 SHUTTLE-BASED LABORATORIES. The prime objective of experiments in shuttle-based laboratories, preferably carried out in sortie missions, is the production of prototype components for subsequent ground testing under practical service conditions. They further permit the manned control and observation of processes. It is, however, recommended to postpone the development of processing equipment until reliable requirements can be defined on the basis of the results of the rocket test program as they will become available in the later part of CY 74. Experiment definition and equipment design studies may then be scheduled for CY 75/76 and hardware fabrication and checkout for CY 77, to meet the presently planned initiation of sortie missions in CY 78.